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ABSTRACT

This document is an instructional module package prepared in objective form for use by an instructor familiar with the spectrophotometric analysis of manganese in water using the persulfate method. Included are objectives, an instructor guide, student handouts, and transparency masters. A video tape is also available from the author. This module considers use of the spectrophotometer, Beer's Law, calibration curves, preparation of reagents and standards, test procedures and interferences.
(Author/RH)

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MANGANESE ANALYSIS IN WATER SAMPLES

Training Module 5.211.2.77

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September, 1977

SE 024 159

Module No:	Module Title: Manganese Analysis in Water Samples
Approx. Time: * 5 or 3.75 hours	Submodule Title: Topic: Summary
Instructional Objective: Upon completion of this module the participant should be able to: 1. Given the proper equipment and reagents, determine the concentration of manganese spectrophotometrically with the Persulfate Method. 2. Describe interferences and possible sources of error in a manganese analysis. 3. Describe safety precautions which should be employed in performing the analysis. 4. Explain why the manganese determination should be performed and how the test results will be used.	
Instructional Aids: 1. Fe/Mn Video tape 2. Overhead Transparencies (Mn1-Mn8) 3. Lab Safety Handout	
Instructional Approach: Lecture, demonstration, discussion, videotape viewing, practical.	
References: 1. W.H.Nebergall, C.Schmidt, H.F.Holtzclaw,Jr., "College Chemistry with Qualitative Analysis." 5th ed., Lexington, Mass. D.C. Heath, 1976. 2. "Standard Methods for the Examination of Water and Wastewater" 14th ed. 3. Operator's Manual for the Bausch and Lomb Spectronic 20 Spectrophotometer.	
Class Assignments: 3 * The module may be shortened to 3.75 hours if used in conjunction with the iron analysis module. In which case, the spectrophotometer	

Module No:	Module Title: Manganese Analysis in Water Samples
Approx. Time:	Submodule Title:
0.5 hours	Topic: Introduction to Manganese Chemistry
Instructional Objective: Upon completion of this module, the participant should be able to: 1. Locate manganese on the periodic table and not confuse it with magnesium. 2. Classify manganese as an element. 3. Identify the major oxidation states of manganese. 4. Name and give the formula of three different compounds of manganese.	
Instructional Aids: Transparency Mnl - periodic chart of the elements.	
Instructional Approach: Lecture/discussion	
References: Nebergall, <u>et al</u> , pp. 895-898	
Class Assignments: None	

Module No:

Mn

Topic:

Introduction to Manganese Chemistry

Instructor Notes:

Instructor Outline:

Transparency Mnl

Periodic Chart

1. The periodic chart lists all the elements in the universe and their symbols. Manganese is one of these. Its symbol is Mn.
2. Manganese by its location next to iron is a transition metal.
3. Oxidation states:
 - a. +4
 - b. +2
 - c. +7
4. Compounds:
Mn⁰² manganese dioxide
MnCl₂ manganese (II) chloride
KMn⁰ potassium permanganate

Module No:	Module Title: Manganese Analysis in Water Samples
	Submodule Title:
Approx. Time: 0.5 hours	Topic: Manganese and Water
Instructional Objective: Upon completion of this module, the participant should be able to: -1. Describe the undesirable effects of manganese in water supplies. 2. Name four ways in which manganese is found in water. 3. Describe how manganese may be removed from water. 4. Differentiate between the permanganate ion, the manganese (II) ion and manganese dioxide. 5. List two methods by which errors in the determination can be prevented in the sampling process. 6. Write a balanced chemical equation which describes the oxidation of manganese (II) ion to permanganate ion with persulfate. 7. Write a balanced chemical equation which describes the oxidation of oxalate with permanganate.	
Instructional Aids: Transparency Mn2 - How oxidation states of manganese are found in water. Transparency Mn3 - REDOX reactions of manganese.	
Instructional Approach: Lecture/demonstration	
References: Standard Methods, p. 224.	
Class Assignments: None	

Module No:	Topic:
Mn	Manganese and Water
Instructor Notes:	Instructor Outline:
Transparency Mn2 REDOX reaction of manganese	- Undesirable effects - taste in water - stains in laundry and plumbing - No more than 1 mg per liter allowed - Manganese found in water as Mn^{2+} MnO_2 suspension - complexed Mn^{3+} MnO_4^- - Removal - pH adjustment, precipitation - aeration - chlorination - Forms Mn^{2+} Manganese (II) MnO_2 insoluble manganese dioxide MnO_4^- permanganate - Elimination of errors - determine soon after collection - acidify immediately following collect $8H_2O + 2Mn^{2+} + 5S_2O_8^{2-} \rightarrow 10SO_4^{2-} + 2MnO_4^- + 16H^+$ $16H^+ + 2MnO_4^- + 5C_2O_4^{2-} \rightarrow 10CO_2 + 2Mn^{2+} + 8H_2O$

Module No:	Module Title: Manganese Analysis in Water Samples
Approx. Time: 0.5 hours	Submodule Title: Topic: Spectrophotometry and the Use of the Spectrophotometer.
Instructional Objective: Upon completion of this module the participant should be able to: 1. Operate a spectrophotometer such as the Bausch and Lomb Spectronic-20: a. turn on and warm-up b. set wavelength c. set zero d. set 100% T 2. Determine the absorbance and transmittance of a colored sample at a pre-determined wavelength. 3. Briefly explain how a spectrophotometer works. 4. Qualitatively, relate absorbance to deepness of color and concentration of solute. 5. Describe how wavelength and hue of color are related.	
Instructional Aids: Transparency Mn4 - condensed spectrophotometer operating instruction Transparency Mn5 - diagram of spectrophotometer.	
Instructional Approach: Lecture/demonstration; practice	
References: Operators manual for spectrophotometer used.	
Class Assignments: Read operator's manual prior to session.	

Module No:

Mn

Topic:

Spectrophotometry

Instructor Notes:

The instructor will need to have a spectrophotometer for demonstration and spectrophotometer(s) for student use.

Transparency Mn5
operating instruction

Colored samples:
dilute KMnO_4 or Mn-phenanthroline at 525 and 510 nm respectively.

Transparency Mn6
spectrophotometer diagram

Instructor Outline:

1. Review operating instructions. have students prepare instrument for warm-up.
2. Determine absorbance of sample - Have students do same.
How the spectrophotometer works.
3. Trace light path through spectrophotometer.
4. Greater absorbance - deeper color, higher concentration.
5. Wavelength determines color of light absorbed, complement transmitted.

Module No:	Module Title: Manganese Analysis in Water Samples
Approx. Time: 0.5 hours	Submodule Title:
	Topic: Beer's law and calibration curves
Instructional Objective: Upon completion of this module the participant should be able to: 1. Draw a Beer's law calibration curve given the appropriate experimental data. 2. Determine the slope of the curve and the value of K in the equation $C=KA$, where C is concentration and A is absorbance. 3. Given the value of A, determine the value of C from a Beer's law curve of from the value of K in (2) above.	
Instructional Aids: Mn6-Graph paper transparency	
Instructional Approach: Lecture/practice	
References: Standard Methods, pp. 10-13, 23-25.	
Class Assignments: None	

Module No:

Mn

Topic:

Beer's Law and Calibration Curves

Instructor Notes:

You will plot the curve on the transparency Mn7 with a ruler the students will follow.

The following data is suggested.

(x axis) conc. (mg/l)	(y axis) absorbance
10.0	0.11
20.	0.19
30.	0.30
40.	0.41
50.	0.49

This will have a slope of 0.01

$$k = 100 \frac{1}{\text{mg}}$$

Instructor Outline:

1. curve
 - a. plot points
 - b. draw best straight line
2. slope $= \frac{\Delta y}{\Delta x}$
 - a. or $\frac{y_1 - y_2}{x_1 - x_2} = \text{slope}$
 - b. $k = \frac{1}{\text{slope}}$
3. determine value of C
 - a. directly from graph.
 - b. by proportions or substitution.

Module No:	Module Title: Manganese Analysis in Water Samples
Approx. Time: 0.75 hours	Submodule Title:
	Topic: Principles of the Persulfate Method of Manganese Analysis
Instructional Objective: Upon completion of the module the participant should be able to: 1. Briefly describe how manganese is determined spectrophotometrically. 2. Describe how all manganese is oxidized to MnO_4^- form by persulfate ion. 3. Describe how solutions of MnO_4^- can be standardized with sodium oxalate solution. 4. Describe the color of the permanganate ion.	
Instructional Aids: Iron/Manganese video tape cassette.	
Instructional Approach: Television; lecture/discussion	
References: Standard Methods, pp. 225-227, 187.	
Class Assignments: None	

Module No: Mn	Topic: Principles of analysis
Instructor Notes:	Instructor Outline:
Show second half of videotape Fe/Mn videotape	1. Show video tape 2. Answer questions concerning video tape 3. Standardization of MnO_4^- weigh out 150 mg $\text{Na}_2\text{C}_2\text{O}_4$, dissolve in 10 ml $9\text{MH}_2\text{SO}_4$, heat, titrate to pink color Normality = $\frac{\text{gNa}_2\text{C}_2\text{O}_4}{(\text{ml to titrate}) \times (0.06701)}$

Module No:	Module Title: Manganese Analysis in Water Samples
Approx. Time: 0.25 hours	Submodule Title: Topic: Lab Safety Review
Instructional Objective: Upon completion of this module the participant should be able to: 1. Locate the following in the laboratory and demonstrate proper use: emergency shower, fire extinguisher, eye wash, first aid kit. 2. Select and use safety glasses, lab coat or apron and gloves in the appropriate situation.	
Instructional Aids: Handout of safety rules for the laboratory.	
Instructional Approach: Lecture/demonstration	
References: Basic Laboratory skills module	
Class Assignments: Read lab safety rules	

Module No:

Mn

Topic:

Lab Safety Review

Instructor Notes:

Instructor Outline:

Hand out Lab Safety Rules
to be determined locally

1. Show the location of and demonstrate
use of various safety devices.
2. Point out the corrosive nature of
concentrated laboratory acids and the
effect of eyes.

Module No:	Module Title: Manganese Analysis in Water Samples
Approx. Time: 0.75 hours	Submodule Title:
	Topic: Preparation of reagents and standards
Instructional Objective: Upon completion of this module the participant should be able to: 1. Prepare all solutions needed for the determination of total manganese. 2. Prepare and standardize 0.1 N KMnO_4 solution. 3. From the standard KMnO_4 solution, prepare a stock solution containing 50mg/l Mn. 4. Calculate the exact concentration of solution in (3) above.	
Instructional Aids: None	
Instructional Approach: Laboratory practice	
References: Standard Methods, P. 225, 226.	
Class Assignments:	

Module No:

Mn

Topic:

Preparation of reagents and standards

Instructor Notes:

Instructor Outline:

1. 75 g H_2SO_4 400 ml HNO_3 200 ml H_2O 200 ml H_3PO_4 .035 g AgNO_3

Dilute to 1 l.

2.a. 3.2 g in 1 l. H_2O ,boil for several
hours, filter
through fritted glass.b. titrate against
150 mg $\text{Na}_2\text{C}_2\text{O}_4$ $N = \frac{g(\text{Na}_2\text{C}_2\text{O}_4)}{150}$ $\frac{(\text{ml to titrate}) \times (}{.06701)}$ $3 \text{ ml} = \frac{4.55}{N}$ add 2 ml H_2SO_4 , 10% NaHSO_3 dropwise, boil,

dilute to 1 l.

1. Reagent Preparation - in groups

Special Reagent

2. Standard KMnO_4 (May be prepared a head and
standardized by students)

a. preparation

b. titrate against $\text{Na}_2\text{C}_2\text{O}_4$ prepare stock KMnO_4 4. If done correctly, "3" will contain
50 mg / l. Mn.

Module No:	Module Title: Manganese Analysis in Water Samples
Approx. Time: 0.75 hours	Submodule Title: Topic: Preparation of Standard Curve and Testing of Sample
Instructional Objective: Upon completion of this module the participant should be able to: 1. Measure aliquots of sample solution and standard solution to fit a standard curve. 2. Process the standard and sample solutions to oxidize the manganese. 3. Dilute all standard and sample solutions to 100 ml properly. 4. Measure the absorbance of the sample and standard solutions at 525 mn and record data. 5. Plot absorbance as a function of total manganese and draw the appropriate straight line. 6. Determine total manganese from the sample absorbance and calibration curve. 7. From total manganese and volume of sample calculate concentration in mg/l Mn.	
Instructional Aids: None	
Instructional Approach: Laboratory practice	
References: Standard Methods, pp. 226, 227.	
Class Assignments: Read Standard Methods pp. 226, 227.	

Module No: Mn	Topic: Preparation of standard curve and testing of sample
Instructor Notes:	Instructor Outline:
suitable unknowns can be prepared from the stock	1. Standard samples a. Add 1 ml; 2 ml, 3 ml, 4 ml, 10 ml; 15 ml, 20 ml, 30 ml, 40 ml to 250 flasks dilute to 50 ml b. Add 5 ml of sample to flask 2. Processing: a. add 5 ml Special Reagent to each flask b. Add water to bring volume to about 90 ml c. add 1 g (NH₄)₂S₂O₈ d. boil and cool 3. Dilute in 100 ml vol. flasks to mark exactly 4. Measure absorbances 5. Plot absorbance of standard vs. total 6. Using absorbance of sample determine total Mn 7. Determine concentration of Mn
mg/l Mn = $\frac{\text{Mg Mn}}{5 \text{ ml sample}}$	

Module No:	Module Title:
	Manganese Analysis in Water Samples
	Submodule Title:
Approx. Time:	Topic:
0.5 hours	Interferences
Instructional Objective: Upon completion of this module the participant should be able to: 1. List various substances or conditions which might interfere with the determination of manganese in water. 2. Describe how to correct for turbidity or interfering color. 3. Describe how MnO_2 may be dissolved to avoid low results. 4. Determine when and how to perform a preliminary digestion with nitric acid. 5. Estimate the accuracy and sensitivity of the method under various conditions.	
Instructional Aids: Mn7 - List of interferences transparency. Mn8 - Diagram of nitric acid digestion transparency.	
Instructional Approach: Lecture/discussion	
References: Standard Methods pp. 224-227, 167-169.	
Class Assignments: None	

Module No:

Mn

Topic:

Interferences

Instructor Notes:

Instructor Outline:

Transparency Mn 7
Interferences

Transparency Mn8
nitric acid digestion

1. Substances which interfere:
 - a. chloride
 - b. organics
 - c. color, turbidity
 - d. air
2. Correction for turbidity:
 - a. make measurement
 - b. "Bleach" with H_2O_2
 - c. read again
 - d. subtract absorbances
3. Dissolve MnO_2 by adding H_2O_2 following special reagent and before boiling
4. Preliminary digestion:
 - a. use in presence of large amounts of organics
 - b. used to break-up organic Mn complexes
5. Accuracy and sensitivity:
 - a. lower limit 5 Mg Mn
 - b. Error: 7.2%

Exam Questions

Manganese Analysis in Water Samples

Introduction to Manganese Chemistry

1. The chemical symbol for manganese is:
 - a. Mg
 - b. Mn
 - c. Hg
 - d. Mo
 2. Manganese is a(n):
 - a. actinide
 - b. lanthanide
 - c. transition metal
 - d. halogen
 3. What is the oxidation number of manganese in the permanganate (MnO_4^-) ion?
 4. The compound KMnO_4 is called _____.
- Manganese and water
5. True or False: Manganese is harmful in water because it causes cancer.
 6. True or False: Manganese can be found suspended in water as MnO_2 .
 7. Name one method used to remove manganese from water.
 8. True or False: The permanganate ion is almost colorless.
 9. If a sample is not analyzed for manganese soon after collection and is exposed to air, how will the results of the determination be affected?
 10. When manganese is oxidized to permanganate, by persulfate, what is the persulfate converted to?
 11. When oxalate is oxidized by permanganate, what are the products?
- Spectrophotometry
12. True or False: Zero percent transmittance is set with distilled water in the sample holder.
 13. True or False: As absorbance increases, transmittance decreases
 14. A spectrophotometer measures _____.
 15. As concentration increases absorbance of a colored sample _____.
 16. Red light has a (shorter, longer) wavelength than blue light.
- Beer's Law
17. True or False: With good Beer's Law data; the plotted line should pass through the origin ($A=0$, $C=0$).
 18. In the equation $C=kA$, if $C=10$ mg/l and $A=.2$, what is the value of k ?
 19. If $A=.2$ and $k=100$ mg/l what would the value of C be in the equation $C=kA$?
- Principles of Analysis
20. Which ion contains the color in the manganese analysis?
 21. Which ion causes the oxidation of manganese?
 22. Permanganate can be standardized by titration against _____.
 23. What color is the permanganate ion?
- Lab Safety
24. Which device in the laboratory can be used to put out fires?
 25. A lab apron is used for what purpose?
- Preparation of Reagents
26. Should safety goggles be worn when the "Special reagent" is prepared?
 27. If it takes 0.5 ml of KMnO_4 solution to titrate a blank solution and 30 ml of KMnO_4 to titrate a solution containing 0.150 g

$\text{Na}_2\text{C}_2\text{O}_4$, what is the normality of the KMnO_4 ? The relevant equation is: $N = (g \text{ Na}_2\text{C}_2\text{O}_4) / ((A-B) \times 0.06701)$.

28. To prepare a solution which contains 50 mg per liter, how many ml of 0.1N KMnO_4 should be diluted to one liter? ml $\text{KMnO}_4 = 4.55/N$
29. If in a solution, 1.00 ml = 50.0 μgMn , what would the concentration of the solution be in mg/l?

Preparation of Standard Curve and Testing of Sample

30. How many ml of KMnO_4 solution are needed to prepare a standard which contains 150 μgMn if 1 ml = 50 μgMn ?
31. How long should the Mn solution boil in the conversion to permanganate step?
- 1 sec.
 - 1 hour
 - 1 min.
 - 30 min.
32. When a volumetric flask is filled to the mark, should the bottom or top of the meniscus be even with the mark?
33. In addition to absorbance what other value should be recorded when the calibration curve is prepared?
34. When absorbance is plotted as a function of total manganese, should the line be curved or straight?
35. True or False: Given a calibration curve of absorbance vs. total Mn the total manganese in a sample of standard volume can be found if the absorbance determined under standard conditions is known.
36. 5 ml of solution are taken for analysis. Calculate the concentration if it is found that 50 μg Mn are present.

Interferences

37. Which of the following in amounts less than 0.1 g will not interfere with the manganese determination?
- iodide
 - air
 - chloride
 - organic matter
38. True or False: Interfering color can be compensated for by "bleaching" the permanganate following analysis and reading the new absorbance and subtracting.
39. How can MnO_2 be redissolved?
40. Preliminary digestion with nitric acid is performed when organic matter is present and when excessive amounts of _____ ion are present.
41. What is the minimum amount of manganese which can be detected with a 1 cm cell in this method?
- 30 micrograms
 - 0.3 micrograms
 - 10 grams
 - 10 milligrams

MANGANESE ANALYSIS IN WATER SAMPLES

EQUIPMENT AND SUPPLIES LIST

EQUIPMENT

- | | |
|-----------------------------|---------------------------------------|
| 1. spectrophotometer | 11. bunsen burner |
| 2. 2 absorbance cells | 12. fritted glass filter, funnel |
| 3. safety goggles | 13. 3 - 400 ml beakers |
| 4. apron | 14. 10 and 100 ml graduated cylinders |
| 5. laboratory balance | 15. thermometer (-10 to 120°C) |
| 6. 1000 ml beaker | 16. 50 ml buret |
| 7. 1 liter volumetric flask | 17. 5 ml and 10 ml pipets |
| 8. ring stand | 18. 6 - 250 ml erlenmeyer flasks |
| 9. iron ring | 19. 100 ml volumetric flask |
| 10. wire gauze | 20. graph paper |

CHEMICALS

1. mercury (II) sulfate
2. concentrated nitric acid
3. 85% phosphoric acid
4. distilled water
5. silver nitrate
6. ammonium persulfate
7. potassium permanganate
8. anhydrous sodium oxalate
9. concentrated sulfuric acid
10. sodium bisulfite
11. 9 m sulfuric acid

Manganese Analysis in Water Samples

Laboratory Procedure

I. Preparation of Standards and Reagents

A. Obtain all the equipment, supplies, and chemicals listed on the "Equipment" hand out. Turn on spectrophotometer for warm-up.

B. Prepare the following solutions:

1. Special reagent: To a 1ℓ volumetric flask add 75 g mercuric sulfate (HgSO_4). Add 400 ml concentrated nitric acid (HNO_3) and 200 ml distilled water. Mix to dissolve. Add to this 200 ml 85% phosphoric acid (H_3PO_4), mix. Add 0.035g silver nitrate AgNO_3 , mix. Allow the solution to cool, and fill the flask to the mark with distilled water.

2. KMnO_4 solution (0.1N): This solution may have been prepared ahead. Add 3.2 g potassium permanganate to a 1ℓ volumetric flask. Dilute to the mark with distilled water. Allow to age in a sunlit window sill for one week or heat for several hours at 90°C . Pour the cooled solution through a scintered glass filter crucible into a vacuum filter flask. Transfer to a 1 liter bottle; label the bottle.

3. Sodium bisulfite solution: Add 10.0 g sodium bisulfite (NaHSO_3) to a 150 ml bottle. Add 100 ml distilled water. Mix to dissolve. Label the bottle.

C. Standardization of KMnO_4 solution.

1. Into three 400 ml beakers, weigh samples, 0.15 g standard, dried, sodium oxalate ($\text{Na}_2\text{C}_2\text{O}_4$). Record the exact weight of each sample to the nearest 0.1 mg. Use an analytical balance.

2. Add 100 ml distilled water. Stir to dissolve. Add 10 ml 9M sulfuric acid (H_2SO_4).

3. Heat each beaker to 90°C .

4. Add KMnO_4 solution from "B" above to 50 ml buret, rinse, fill buret.

5. For each beaker, titrate the warm solution rapidly until a faint pink color persists for a minute. Record the initial and final values of the buret. Subtract the initial from the final. The buret may need to be refilled several times.

6. Determine the normality of each sample from the formula:

$$N(\text{KMnO}_4) = (\text{grams Na}_2\text{C}_2\text{O}_4) / (\text{ml KMnO}_4) \times 0.6701$$

7. Add the three normalities from "6" and divide by 3 for the average normality.

8. Record the average normality on the KMnO_4 bottle label.

- D. Preparation of Standard KMnO_4 (50 $\mu\text{g}/\text{ml}$) solution.
1. Determine the number of ml KMnO_4 solution needed to prepare a standard solution from the formula: $\text{ml} = 4.55 / [N(\text{KMnO}_4)]$
 2. Add (from the buret) the number of ml KMnO_4 solution calculated above to a 150 ml beaker.
 3. To the beaker add 2 ml concentrated H_2SO_4 .
 4. With stirring add drop-by-drop the sodium bisulfite solution until the color disappears.
 5. Boil the solution. (Wear safety glasses). Cool the solution and transfer to a 1 liter volumetric flask. Rinse into the flask with distilled water and dilute to the mark. Mix by inversion.

II. Preparation of sample and standards for calibration curve.

- A. Obtain a sample for analysis. A well-mixed sample should be chosen. Analysis should begin as soon as possible after collection. If the sample is to be stored, 1.0ml concentrated HCl should be added to the sample.
- B. Label six 250 ml erlynmeyer flasks as follows: Sample, 50 μg , 100 μg , 200 μg , 400 μg , 500 μg . Pipet into each flask, respectively, 5.0 ml sample, 1.0 ml standard, 2.0 ml standard, 4.0 ml standard, 8.0 ml standard and 10 ml standard. Dilute each sample to about 50 ml with distilled water, mix. Add to each flask 5 ml special reagent and 35 ml distilled water. Mix. Add to each flask 1g ammonium persulfate $(\text{NH}_4)_2\text{S}_2\text{O}_8$. Bring each flask to a boil and boil for one minute. Cool each flask to room temperature by allowing each to stand for 1 minute and then by running tap water over each.
- C. Label six 100 ml volumetric flasks in a manner similar to the erlynmeyer flasks in part B. Transfer the contents of each erlynmeyer flask to the appropriate volumetric flask. The erlynmeyer flasks should be rinsed with distilled water. The rinsings should flow into the volumetric flasks. Fill the volumetric flasks to the mark with distilled water. Mix by inversion.

III. Spectrophotometric measurement

- A. Set the spectrophotometer wavelength to 525 nm. Set 0% T with the sample compartment empty. Fill a 1 cm absorption cell with distilled water, insert into the sample compartment, and set 100% T. Transfer contents of the volumetric flask to absorption cells, insert each cell into the sample compartment and record the absorbance of each sample.
- B. Label a piece of graph paper: x axis - $\mu\text{g Mn}$, y axis - absorbance; insert values 0-1.0 on the y axis, 0-500 on the x axis. Plot the data points: Absorbance as a function of $\mu\text{g Mn}$. With a straight-edge, draw the best straight line through the origin and the points. Title the graph: "Beer's Law Calibration Curve for Manganese."

- C. Find the point at which the calibration line crosses the sample absorbance value. Determine the " $\mu\text{g Mn}$ " corresponding to this value. Record the μg Manganese.
- D. Determine the manganese concentration by dividing " $\mu\text{g Mn}$ " by 5.0. This is the manganese concentration in mg/l .
- E. Comment on possible sources of error or other unusual problems in the analysis including sample collection, storage, precipitation or pre-treatment.

Data Sheet for Manganese Analysis

Sample Number _____

I. Standardization of KMnO_4 solution.

Beaker	I	II	III
Mass of beaker + $\text{Na}_2\text{C}_2\text{O}_4$	_____ g	_____ g	_____ g
Mass of beaker	_____ g	_____ g	_____ g
Mass of $\text{Na}_2\text{C}_2\text{O}_4$	_____ g	_____ g	_____ g
Initial buret reading	_____ ml	_____ ml	_____ ml
Final buret reading	_____ ml	_____ ml	_____ ml
ml KMnO_4 solution	_____ ml	_____ ml	_____ ml

Normality: $\frac{\text{gNa}_2\text{C}_2\text{O}_4}{\text{mlKMnO}_4 \times .06701}$ _____ N _____ N _____ N

Average Normality $\frac{\text{I} + \text{II} + \text{III}}{3.0} =$ _____ N

ml KMnO_4 needed for 50 $\mu\text{g/ml}$ solution:
 $4.55/\text{N}(\text{KMnO}_4) =$ _____ ml

Spectrophotometric measurement:

flask label	ml added	absorbance
sample	5.0 sample	_____
50 mg	1.0	_____
100 mg	2.0	_____
200 mg	4.0	_____
400 mg	8.0	_____
500 mg	10.0	_____

Micrograms manganese _____

Manganese concentration = $\frac{\mu\text{gMn}}{5.0}$ _____ mg/l

Comments:

Analyst: _____

Date _____

PERIODIC TABLE OF THE ELEMENTS

Atomic weight values listed in parentheses are approximate.

TRANSPARENCY MN2

How Manganese is found in Water

oxidation state	form
II	Mn^{2+}
III	complexed Mn^{3+}
IV	MnO_2 suspension
VII	MnO_4^- , permanganate

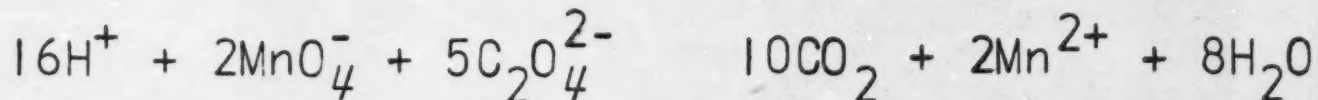
TRANSPARENCY MN3

REDOX Reactions of Manganese

I. With persulfate



II. With oxalate

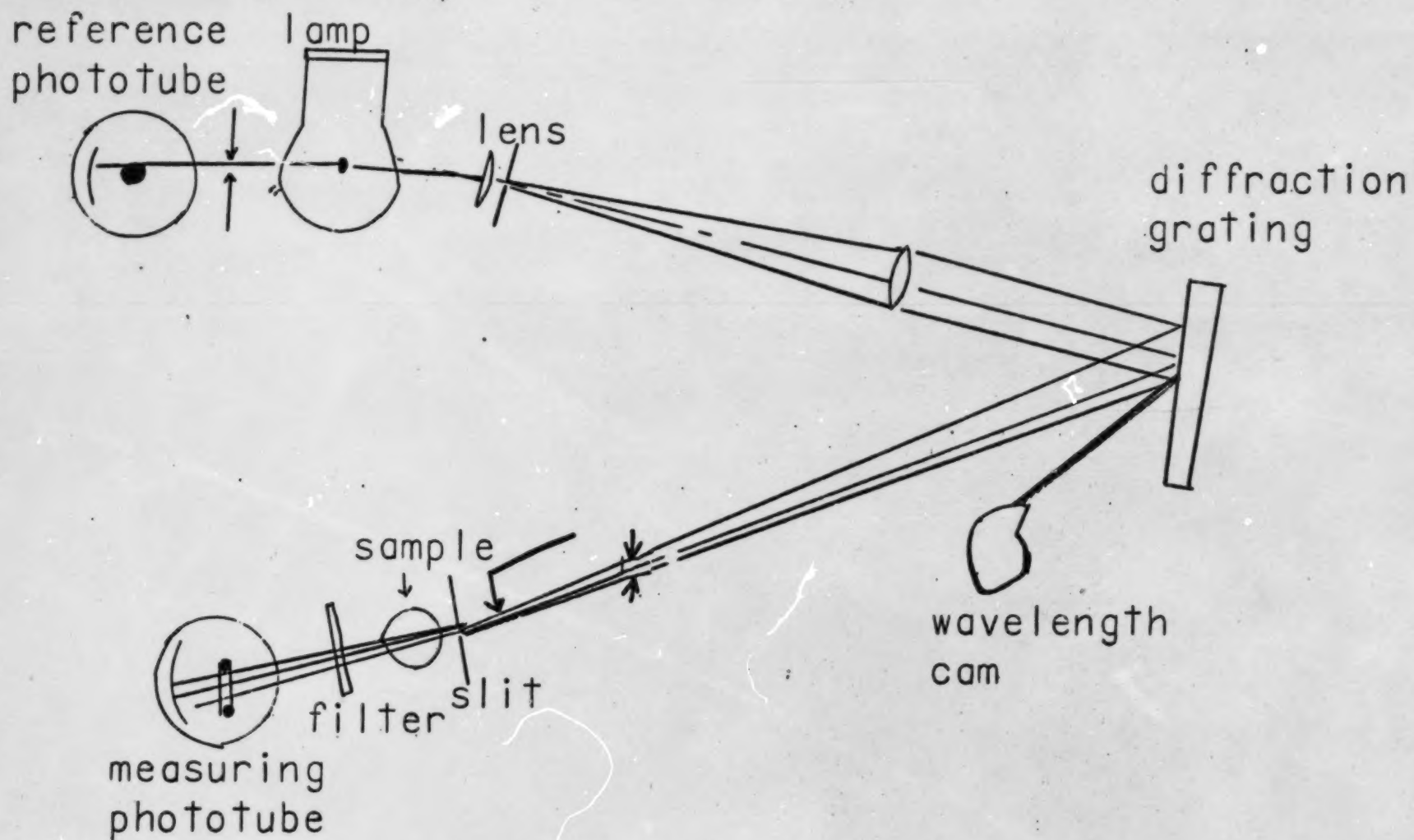


TRANSPARENCY MN4

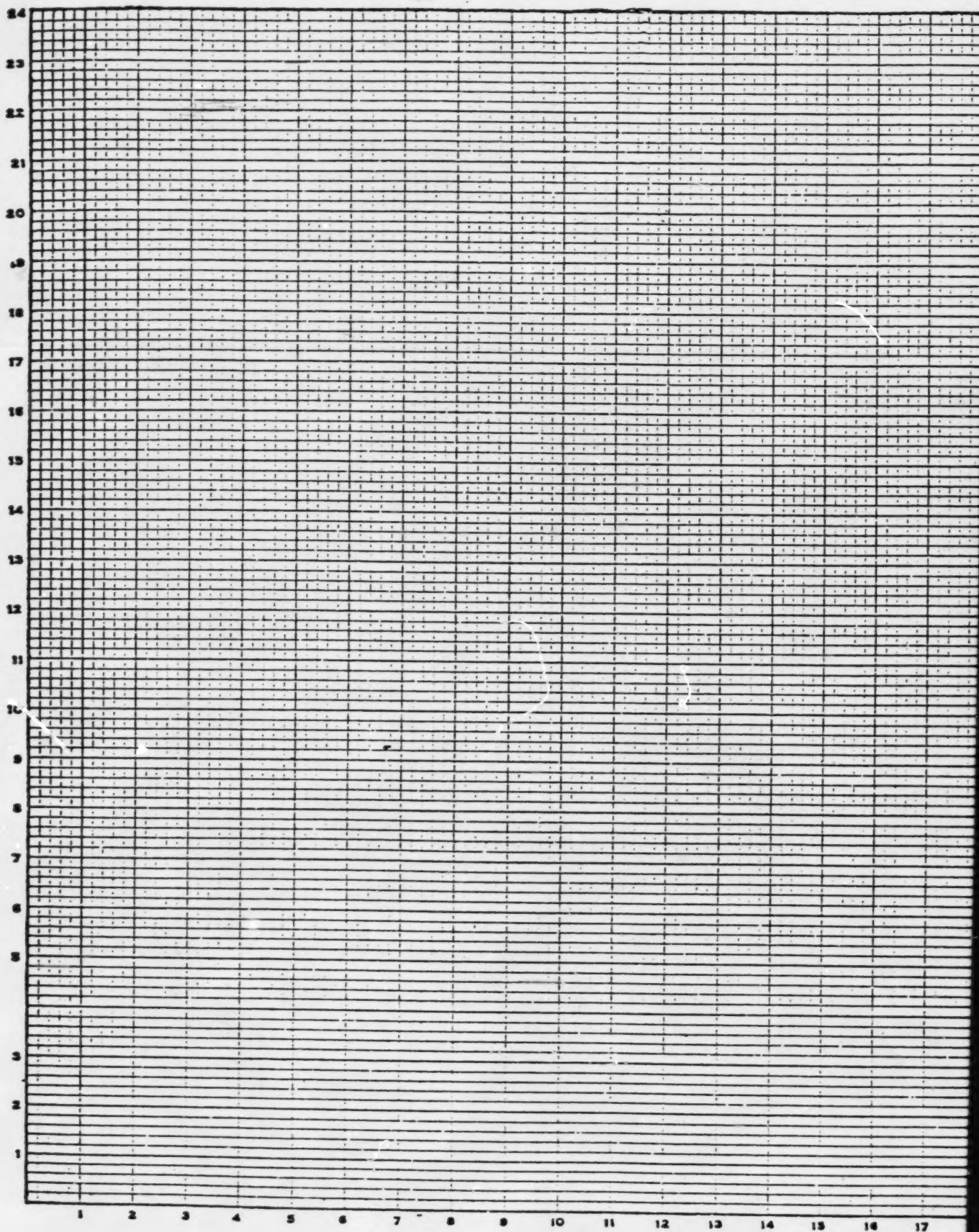
Condensed Spectrophotometer Operating instructions

1. Turn on- warmup 15 min.
knob left front
2. Set zero
knob left front
3. Set wavelength
knob top right
4. Insert blank
sample compartment
5. Set 100% T - full scale
knob right front
6. Insert unknown
sample compartment
7. Read absorbance or %T

TRANSPARENCY MN5
Spectrophotometer diagram



Graph paper for Calibration Curve



TRANSPARENCY MN7

Interferences

Interfering substance	Correction
1. Chloride	HgSO_4
2. Organics	Nitric acid Digestion
3. Turbidity, color.	"Bleach" with H_2O_2 following measure, subtract background
4. Air	30% H_2O_2 after special reagent

TRANSPARENCY MN8

Nitric Acid Digestion

